Nitrate accelerated corrosion of lead solder in potable water systems

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abstract

Although nitrate is generally believed to have little effect on lead corrosion, bench-scale tests in this work revealed that increasing nitrate in the range of occurrence in potable water (0–10 mg/L N) can dramatically increase lead leaching from simulated soldered pipe joints. Lead in water created slightly increased galvanic currents between solder and copper pipe, but nitrate also altered the nature of the attack in a manner that caused solder particles to detach into the water. Chloramine decay and the associated conversion of ammonia to nitrate could create much higher lead contamination of potable water from solder in some cases.

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1. Introduction

Nitrate (NO_3^-) concentrations in drinking water are increasing in some areas of the country due to fertilizer run-off, release of other nitrogen containing compounds from industry, and nitrification in chloraminated systems [1–4]. The US Environmental Protection Agency (USEPA) has established a maximum contaminant level (MCL) of 10 mg/L NO_3–N due to concern over adverse health effects [5,6]. While many water supplies contain less than 0.1 mg/L NO_3–N, exceedances of the nitrate MCL are not uncommon and may be increasing [7].

It is commonly believed that nitrate generally has little or no effect on corrosion of pure lead pipe or lead solder [8]. However, prior studies of galvanic corrosion due to the physical connection between lead solder and copper pipe noted that the surface potential was very unstable in water with greater than 1 mg/L NO_3–N [9]. Although Gregory [9] did not measure the effects of nitrate on metals release, follow-up research by Dudi [10] noted a significant increase in lead release from leaded brass when 10 mg/L NO_3–N was dosed. Higher lead leaching has also been reported after nitrifying bacteria reduced pH and converted ammonia to nitrate [4,11–13]. However, Zhang et al. [14] determined that the adverse effect of nitrification for pure lead pipes was attributable to the reduced pH instead of the formation of nitrate from ammonia.

Other work on lead corrosion in water with high pH and high nitrate concentrations found that nitrate contributed to the breakdown of passivating scale and created non-uniform corrosion. El Rehim and Mohamed [15] and Amin and Abdel Rehim [16] noted that increasing nitrate concentrations, albeit at levels far higher

than drinking water (0–3.5 g/L NO_3–N), increased the current density in electrochemical cells with lead anodes. Those authors and others also observed more pitting corrosion on lead [15,16] and tin surfaces [17] with higher nitrate concentrations, but increasing alkalinity and phosphate mitigated the nitrate induced pitting to some extent [16,18]. This is consistent with other work where phosphate was effective in inhibiting lead corrosion [9,19–22]. Additionally, higher alkalinity is believed to buffer pH drops that occur during galvanic corrosion at lead anode surfaces [10], and more alkaline pH values generally decrease lead solubility [21,23,24].

In light of the observations regarding potential adverse impacts of nitrate on aspects of lead corrosion, this work was designed to: (1) determine if changes in nitrate in the range commonly encountered in potable water (0–10 mg/L) could adversely affect lead corrosion and lead contamination of potable water; and (2) isolate possible abiotic impacts of nitrification on lead leaching from soldered joints in systems practicing chloramination. Because Zhang et al. [14] confirmed the conventional wisdom that slight changes in nitrate (0–2 mg/L N) had little or no effect on lead leaching from lead pipe alone, potential impacts on galvanic corrosion of lead solder:copper joints was a focus of this research.

2. Materials and methods

2.1. Experimental apparatus

2.1.1. Macrocells

The simulated copper pipe joint macrocells were designed to allow measurement of the galvanic current between 50:50 Pb–Sn solder and copper. The macrocell was constructed with 50:50 Pb–Sn solder wire and copper pipes as described elsewhere [25]. The copper-to-solder ratio was approximately 34:1.

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2.1.2. Solder coupons

The second apparatus was a galvanic lead solder:copper coupon simulating lead solder in a typical copper pipe joint in a home, which was assembled by melting a 25-mm length of 3-mm diameter 50:50 Pb–Sn solder to a 25-mm length of 13-mm diameter copper pipe coupling [26]. The approximate solder-to-copper wetted surface area was 1:7.3. The solder coupons were placed in glass containers and exposed to 100 mL of each test water, and each condition was tested with at least three replicates.

2.2. Water chemistry and testing sequence

Work was conducted in four phases. Phases 1 and 2 were carried out using macrocells, and Phases 3 and 4 were conducted using solder coupons. Test water in Phases 1 through 3 had 24 mg/L alkalinity as $CaCO_3$ (from $NaHCO_3$), 21 ± 1 mg/L Cl^- (from $CaCl_2 \cdot 2H_2O$), 45 ± 3 mg/L SO_4^{2-} (from $CaSO_4 \cdot 2H_2O$ and $MgSO_4$), and a chloride-to-sulphate mass ratio (CSMR) of about 0.47 (Table 1). Similar water was used in Phase 4, but the chloride concentration was increased in some water conditions to represent chloramine decay in a distribution system (Tables 2 and 3).

When chloramine disinfectant was added in Phases 2 through 4, a 4:1 mg Cl_2/mg N ratio was used. Nitrate was added as $NaNO_3$, and all chemicals were added as reagent grade salts to distilled and deionised water. The pH of all conditions was adjusted to 7.7 ± 0.1 by adding CO_2 before placing the water in the test cells.

2.2.1. Phase 1

The macrocells were exposed to approximately 110 mL of each test water (Table 1), and water was changed using a static "dump-and-fill" method [27].

2.2.2. Phase 2

Waters with 0, 1, 2.5, and 10 mg/L NO_3–N were tested in triplicate with macrocells for a period of 10 days in Phase 2 (Table 1), and no disinfectant was added. The conductivity ranged from 172 to 311 μS due to differences in the nitrate concentration (Table 1). The water was changed three times (every 3 or 4 days) using a "dump-and-fill" method [27]. Triplicate samples were collected, and each sample was analyzed for lead.

2.2.3. Phase 3

This phase of the study tested whether increasing nitrate from 0 to 10 mg/L NO_3–N increased lead release from solder coupons. Water with 0, 1, 2.5, 5, and 10 mg/L NO_3–N (Table 1) were selected to represent the typical range found in drinking water and was disinfected with chloramine (3.5 mg/L Cl_2). The test waters were exposed to solder coupons in five replicates for 9 weeks. The water was changed using a static "dump-and-fill" protocol [27] twice per week (Monday and Thursday) to simulate "worst-case" 3-day stagnation conditions encountered in school plumbing systems over weekends. At Week 9 after the lead concentration did not decrease significantly with time, which suggested that the system had

reached pseudo steady-state, individual samples were collected from each of the five replicates to indicate the relative error among the replicates.

2.2.4. Phase 4

The simulated abiotic effect of nitrification in a system using chloramine disinfectant was tested in Phase 4 with solder coupons in triplicate. The test waters were selected to simulate the extreme change in nitrogen chemistry that could occur as water leaves a treatment plant and passes through a distribution system. That is, chloramine (NH_2Cl) first decays to chloride and ammonia (NH_3), followed by conversion of ammonia to nitrite (NO_2^-) and nitrate (Table 2).

To examine a "worst-case" scenario based on results from Phase 3, where the corrosive nitrate threshold occurred with 2.5 mg/L NO_3–N, all water in Phase 4 were dosed with 1.25 mg/L NO_3–N. Therefore, if nitrification occurred in water with 4 mg/L chloramine as Cl_2 and 1 mg/L NH_3–N (4:1 ratio of mg Cl_2/mg NH_3–N), 1 mg/L NO_3–N would form (or a total of 2.25 mg/L NO_3–N) and likely would be corrosive to lead solder. The six water conditions evaluated in Phase 4 were designed to simulate the extremes that could be encountered in a groundwater system with chlorine, chloramine, and various stages of chloramine decay/nitrification, but at a constant pH of 7.7 to isolate changes in ammonia speciation (Table 3).

After Week 6 of Phase 4, the nitrate concentration was increased by 3.75 mg/L NO_3–N for all of the water conditions (Table 3), increasing the nitrate concentration to at least 5 mg/L NO_3–N for all water.

2.3. Analytical methods

Collected samples after exposure to the macrocells or solder coupons were acidified with 2% nitric acid for at least 24 h to dissolve metals and analyzed with an inductively coupled plasma mass spectrometer (ICP-MS) according to Standard Method 3125-B [28]. The pH in the bulk water was measured with an electrode according to Standard Method 4500-H^+ [28]. Ammonia was measured using the salicylate method on a Hach DR 2700 spectrophotometer, according to Standard Method 4500-NH_3 [28]. Total and free chlorine were measured on a Hach DR 2700 spectrophotometer in accordance with Standard Method 4500-Cl [28]. Nitrate and nitrite were measured using DIONEX DX-120 ion chromatography, according to Standard Method 4110 [28]. Galvanic current measurements between the copper pipes and solder in the macrocells were conducted using RadioShack multimeters with 100 Ω resistance. In the convention of this work, negative currents indicated that the lead solder was the anode and was being sacrificed while the copper pipe was the cathode and was being protected.

3. Results and discussion

3.1. Mechanistic study

The magnitude of the galvanic current between the solder and copper pipe increased from approximately 20 to 35 μA when the nitrate (NO_3^-) concentration increased from 0 to 10 mg/L NO_3–N (Fig. 1). This doubling in the amount of current between the lead solder and copper could be due to the higher nitrate concentration or the concurrent doubling in the conductivity [8]. A two-tailed t-test confirmed the relationship between higher nitrate (or higher conductivity) and the higher galvanic current with greater than 95% confidence.

The trend in galvanic currents in Phase 1 was confirmed by lead in water measurements in Phase 2. The lead released from lead sol-

Table 1
Summary of water quality used in Phases 1, 2, and 3.

Parameter	Target nitrate (mg/L NO_3–N)				
	0	1	2.5	5	10
Actual nitrate (mg/L NO_3–N)	0	0.93	2.4	5	11
Chloride (mg/L Cl^-)	22	21	21	21	21
Sulphate (mg/L SO_4^{2-})	46	48	43	44	45
CSMR[a]	0.47	0.44	0.49	0.49	0.47
Alkalinity (mg/L as $CaCO_3$)	24	24	24	24	24
Conductivity (μS)	172	177	251	273	314

[a] Chloride-to-sulphate mass ratio (CSMR).

Table 2
Summary of reactions and effects of chloramine decay on chloride, nitrate, and nitrite concentrations.

Reaction	Balanced equation	Key impact
Reaction 1	$3NH_2Cl \; ? \; N_2 + N H_5 + 3Cl^{ffi} + 3 H^+$	Increase in chloride (2 mg/L Cl^{ffi} per 1 mg/L Cl_2) and formation of ammonia
Reaction 2	$NH_4^+ + 1.9O_2 + 0.069CO_2 + 0.0172HCO_3^{ffi} \; ? \; 0.0172C_5H_7O_2N + 0.983NO_2 + 0.966H_2O + 1.97H^+$	Increase in nitrite (1 mg/L NO_2–N per 1 mg/L NH_3–N)
Reaction 3	$NO_2 + 0.00875NH_4^+ + 0.035CO_2 + 0.00875HCO_3^{ffi} + 0.456O_2 + 0.00875H_2O \; ? \; 0.00875C_5H_7O_2 + N O_3^{ffi}$	Increase in nitrate (1 mg/L NO_3–N per 1 mg/L 1 mg/L NO_2–N)

Refs. [4,34,35].

Table 3
Summary of water conditions in Phase 4.

Parameter	Water condition					
	Control[a]	Free Cl_2[b]	NH_2Cl[c]	Cl^{ffi} + N H_5[d]	Cl^{ffi} + N $O_2^{ffi e}$	Cl^{ffi} + N $O_3^{ffi f}$
Target nitrate (mg/L NO_3–N)	1.25	1.25	1.25	1.25	1.25	2.25
Target nitrite (mg/L NO_2–N)	0	0	0	0	1	0
Target total chlorine (mg/L Cl_2)	0	1	4	0	0	0
Target ammonia (mg/L NH_3–N)	0	0	1	1	0	0
Chloride prior to disinfectant decay (mg/L Cl)	21	21	21	28	28	28
Sulphate (mg/L SO_4^{2ffi})	42	41	41	41	40	41
CSMR[g]	0.50	0.51	0.51	0.68	0.70	0.68
Conductivity (IS)	192	204	223	235	243	241
Conductivity after increase[h] (IS)	283	292	311	323	332	332

[a] Control water was the 1.25 mg/L NO_3–N from Table 1.
[b] Control water dosed with 1 mg/L free chlorine disinfectant.
[c] Control water dosed with chloramine disinfectant (4 mg/L as Cl_2), 4:1 mg Cl_2/mg NH_3–N ratio.
[d] Chloraminated control water after Reaction 1 (Table 2).
[e] Chloraminated control water after Reaction 2 converted to nitrite (Reaction 1 + Reaction 2; Table 2).
[f] Chloraminated control water after Reaction 3 (Table 2).
[g] Chloride-to-sulphate mass ratio (CSMR).
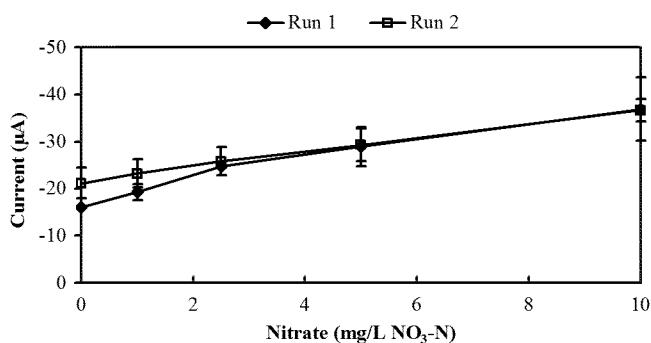[h] After 6 weeks, base-level nitrate was increased from 1.25 to 5 mg/L NO_3–N.

Fig. 1. Impact of increasing nitrate in macrocells on galvanic current in Phase 1. Error bars represent 95% confidence intervals. Negative currents indicate that lead solder was sacrificed and that the copper pipe was protected.
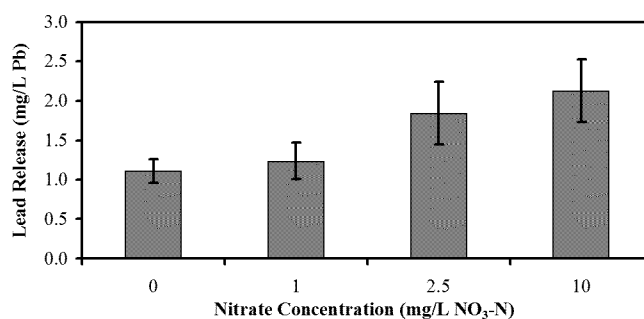

Fig. 2. Lead in water after 3–4 days of stagnation as a function of the nitrate concentration in macrocells in Phase 2. Error bars represent 95% confidence intervals.

der in the macrocells increased nearly 2 times from 1.1 to 2.1 mg/L Pb when nitrate increased from 0 to 10 mg/L N (Fig. 2), and tin release followed similar trends as lead. The impact of higher nitrate was relatively low in these short-term tests with separate anode and cathode arrangements, and follow-up testing used more realistic simulations of lead solder:copper joints using solder coupons.

3.2. Effect of nitrate on galvanic lead solder coupons

Essential trace nutrients to inhibit nitrification [29,30] were not added to the water, and a nitrogen mass balance (i.e., nitrate, nitrite, and ammonia) in the third phase of testing indicated no significant changes in nitrite, total ammonia, or nitrate during the tests after accounting for trace residual nitrate from glassware due to nitric acid washing (Table 4). Thus, significant biological activity was not occurring.

Increasing the nitrate concentration from 0 to 10 mg/L NO_3–N dramatically increased lead leaching from solder coupons. Consistent with expectations, the condition with 0 mg/L NO_3–N had the lowest lead release with 18 Ig/L Pb at Week 9 (Fig. 3). When the nitrate concentration was increased from 0 to 1 mg/L NO_3–N, lead release to the water increased by 2 times to 29 Ig/L Pb. Moreover, increasing nitrate to 2.5 mg/L NO_3–N increased lead in water to 3500 Ig/L Pb, or 194 times relative to water with no nitrate. Further increases in nitrate had lower impact on lead corrosion, but greater nitrate concentrations still increased the average lead release (although the differences were not statistically different). Compared to Week 2, these lead concentrations for water containing at least 2.5 mg/L NO_3–N resulted in at least 3 times more lead at Week 9 than at Week 2, suggesting that the impact of higher nitrate concentrations could worsen with time. In contrast, the conditions with low nitrate (0–1 mg/L NO_3–N) had about 65–80% less lead in water at Week 9 than Week 2. T-tests with triplicate data

Table 4
Nitrate and ammonia values measured before and after a stagnation period during Phase 3. Nitrite values were always below detection (<0.1 mg/L NO_2–N).

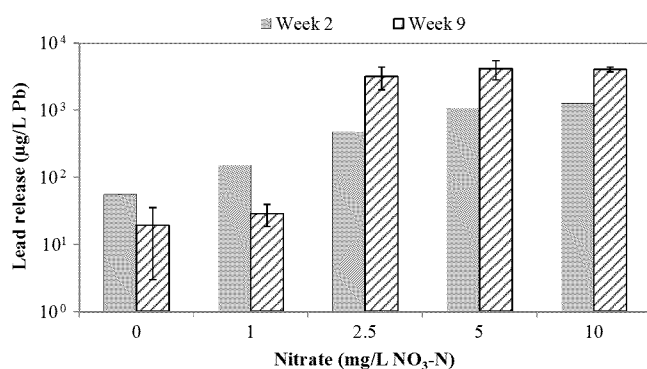| Target nitrate (mg/L NO_3–N)	Nitrate (mg/L NO_3–N)		Ammonia (mg/L NH_3–N)	
	Before	After	Before	After
0	0.00	0.24	0.89	0.82
1	0.93	1.66	0.89	0.80
2.5	2.37	2.58	0.91	0.78
5	4.98	5.59	0.88	0.82
10	10.68	11.03	0.92	0.80

Fig. 3. Effect of increasing nitrate on lead release from solder coupons in Phase 3. Results are shown from Week 2 and Week 9 of the study. Error bars represent 95% confidence intervals for Week 9.

from Week 9 confirmed that the highest three nitrate conditions (2.5, 5, and 10 mg/L NO_3–N) released significantly more lead with greater than 95% confidence than the lower two nitrate conditions (0 and 1 mg/L NO_3–N). Similar trends were observed for tin release, although much less tin compared to lead was released to the water (3 µg/L Sn per 10 µg/L Pb).

Increasing the conductivity by adding nitrate to the water can explain some of the increases in galvanic lead corrosion [8]. However, a parallel study with the same water but with chloride (from NaCl) instead of nitrate added to the water found much lower lead release for the same conductivity (results not shown). Specifically, lead release in water with the same conductivity as the test water with 10 mg/L NO_3–N (311 µS) had 32 times less lead or 140 µg/L Pb at Week 9 (results not shown) compared to the 10 mg/L NO_3–N water. That is, nitrate was more corrosive than chloride for this water type, and the much increased lead contamination of the water from nitrate was not explained by higher conductivity alone.

Visually, the solder coupons exposed to 10 mg/L NO_3–N in Phase 3 were corroded to a much greater extent than solder coupons without nitrate (Fig. 4). Moreover, the water with more
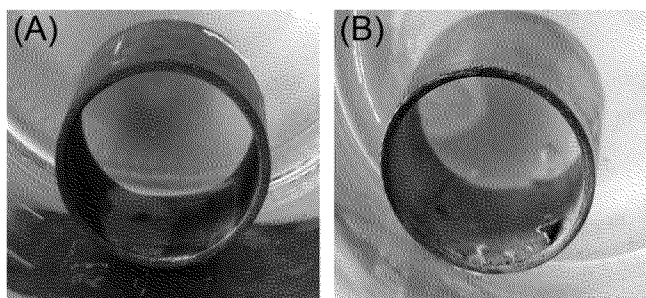
nitrate caused pieces of the lead solder to peel away from the surface of the copper coupling, and the surface of the solder became non-uniformly corroded. In some cases, pieces of metallic solder were observed to detach from the solder coupon into the water. Magnified images showed that the solder surface exposed to no nitrate was virtually unchanged during the test, whereas layers of the solder surface exposed to 10 mg/L NO_3–N were subjected to severe non-uniform corrosion (Fig. 5). The measurements of lead in water confirmed the visual observations of aggressive attack on the lead solder. These results and visual observations suggest that the severe corrosive attack was triggered when more than about 2.5 mg/L NO_3–N was present in this water.

It is possible that the 50:50 Pb–Sn solder was attacked through intergranular corrosion, which is a well-known mechanism for nitrate-induced corrosion of steel and silver [31,32]. Nitrate was not part of the cathodic reaction because the nitrate concentration did not decrease after stagnation (Table 4), which was a mechanism observed by others in the corrosion of tin in low pH (3.5) canned food [33].

Clearly the impacts of nitrate in the longer-term testing with solder coupons were dramatically worse than observed in testing with the macrocell (Phases 1 and 2). Specifically, solder coupons exposed to 2.5 mg/L NO_3–N water had 8.6 times more lead leaching at Week 2 than the control water without nitrate (Fig. 3). In contrast, the same change in the nitrate concentration in the macrocell test resulted in only 1.7 times more lead release and galvanic current (Fig. 2). The effect of nitrate appears to be attributable to a highly localized attack on the lead solder, which caused pieces of solder to fall into the water, because relatively slight increases in galvanic current due to the nitrate (or concomitant increase in conductivity) were observed in the macrocell arrangement. Other possible explanations for the discrepancy include: (1) differences due to melting of the solder in the solder coupon, versus using unmelted Pb/Sn solder wire in the macrocell, (2) larger physical distance between the lead solder wire and the copper pipe cathode in the macrocell (½ cm path length through water) compared to the simulated joint (direct connection of 0 cm). In any case, results

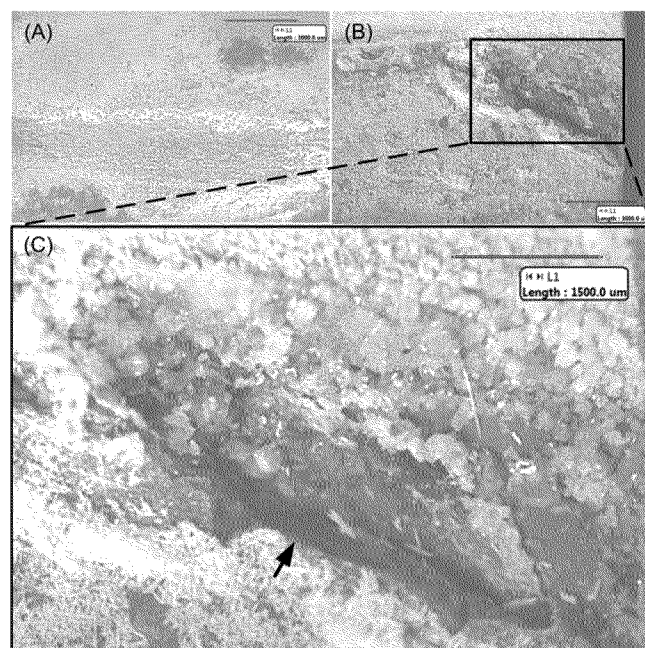Fig. 5. Microscope images captured of the 50:50 Pb–Sn solder surface after 10 weeks of exposure to (A) 0 mg/L NO_3–N and (B, C) 10 mg/L NO_3–N in Phase 3. Image (C) is a 2^j magnification of Image (B). The pictures were captured approximately 6 months after the coupons were removed from the water and dried.

Fig. 4. Galvanic lead solder coupons after 6 weeks of exposure to (A) 0 mg/L NO_3–N and (B) 10 mg/L NO_3–N in Phase 3.

suggest that in actual joints found in a potable water system, the nitrate effect could sometimes be severe.

3.3. Effect of disinfectants and simulated nitrification on lead release

The impacts observed in Fig. 3 raised serious concerns about whether changes in nitrogen speciation occurring in systems using chloramine disinfectant could affect lead leaching from solder in homes. A series of tests was designed to simulate biological consequences of nitrification by examining abiotic changes in nitrogen chemistry that can occur in a distribution system as water leaves a treatment plant and passes through a distribution system [4]. That is, chloramine (NH_2Cl) first decays to chloride (Cl^{ffi}) and ammonia (NH_3), followed by conversion of ammonia to nitrite (NO_2^{ffi}) and nitrate (Table 2). These simulated reactions were evaluated at two levels of nitrate: (1) less than 2.5 mg/L N, and (2) greater than 5 mg/L N.

3.3.1. Conditions with less than 2.5 mg/L NO₃–N

The control condition, which had 1.25 mg/L NO_3–N, released 110 lg/L Pb from the solder coupons (Fig. 6). Disinfectant dosing of either free chlorine or chloramine significantly decreased lead leaching relative to the control water with no disinfectant. Specifically, dosing 1 mg/L free chlorine disinfectant (as Cl_2) decreased lead leaching by about 50%, whereas 4 mg/L chloramine (as Cl_2) decreased lead leaching by about 75% (Fig. 6). During stagnation in this bench-scale test, the free chlorine decayed 100% to chloride, whereas the chloramine decayed about 90%.

In the test simulating complete decay of chloramine to Cl^{ffi} and NH_3, lead leaching increased to about 190 lg/L Pb, or 1.7 times more than the control condition and 7.2 times more than the chloramine condition. In the condition representing complete conversion of chloramine to NO_3^{ffi} and Cl^{ffi}, lead leaching increased to 520 lg/L Pb, which was 4.7 times more lead than the control condition, and 20 times more lead than the chloramine condition (Fig. 6). Interestingly, the condition simulating complete conversion of chloramine to Cl^{ffi} and NO_2^{ffi} was similar to the control condition, but was about 3.7 times higher than the chloramine condition (Fig. 6).

T-tests confirmed all the trends with greater than 95% confidence with Bonferroni correction. The results illustrate that even without accounting for the potentially important impacts of reduced pH due to nitrification [14], corrosivity of water to solder in chloraminated systems can change markedly as it passes through the distribution system, especially if nitrification occurs with formation of nitrate (Fig. 6). This has important consequences for selection of sampling sites for Lead and Copper Rule Compliance and for understanding human exposure to elevated lead.
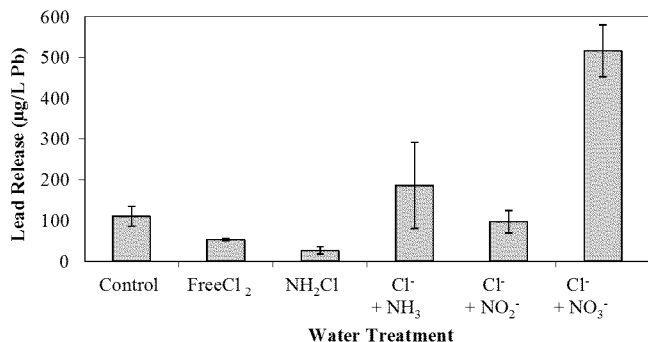

Fig. 6. The effect of disinfectant decay on lead release from solder coupons in Phase 4. The simulated joints were exposed to test waters in Table 3. Error bars represent 95% confidence. Results are shown from Week 6 of Phase 4.
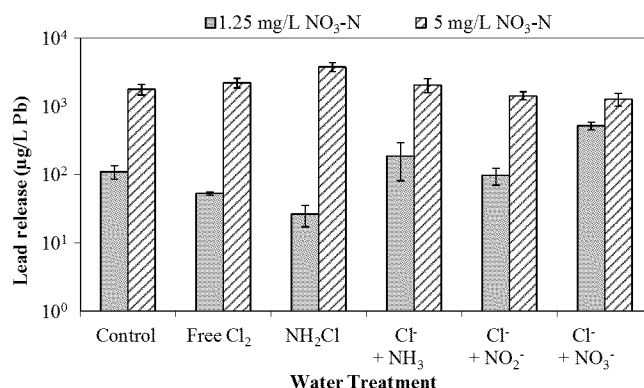

Fig. 7. The effect of increasing base-level nitrate from 1.25 to 5 mg/L NO₃–N on lead release from solder coupons in Phase 4. Error bars represent 95% confidence intervals. Light coloured bars show lead release prior to nitrate increase at Week 6 of the study, and striped bars are lead release 2 weeks after nitrate was increased in all water (Week 8).

3.3.2. Conditions with greater than 5 mg/L NO₃–N

To examine impacts of chloramine decay and nitrification in a system starting with a higher concentration of nitrate (Table 3), nitrate was raised to a base level of 5 mg/L NO₃–N 6 weeks into Phase 4. Within 1 week, lead leaching increased by 2–12 times for all conditions due to the greater nitrate concentration (results not shown). Moreover, the lead release increased 2.5–144 times after 2 weeks at the higher nitrate concentration compared to the results at less than 2.25 mg/L NO₃–N (Fig. 7). The previous trends observed at lower concentrations of nitrate were qualitatively different. Specifically, for the system with greater than 5 mg/L NO₃–N, water with chloramine had the highest lead, whereas the condition simulating complete conversion to nitrate had the lowest lead release (Fig. 7), which is completely different from results at the lower nitrate level (Fig. 6). This difference may be explained by the non-uniform nature of the nitrate attack on lead solder in these higher nitrate conditions (>5 mg/L NO₃–N). Furthermore, there was enough variability in lead release that only the chloramine and free chorine conditions were significantly different from the other conditions with 95% confidence and after applying a Bonferroni correction. Tin leaching from solder followed similar trends as lead (results not shown). These observations reinforces the findings of Dudi [10], who discovered that nitrate caused very erratic lead leaching from brass.

4. Conclusions

(1) There was an increase in galvanic current with greater nitrate concentrations in short-term tests with macrocells, which also corresponded to a doubling of conductivity and lead leaching.

(2) Increasing the nitrate concentration in water from a low level (0–1 mg/L NO₃–N) to a high level (2.5–10 mg/L NO₃–N) could result in dramatic increases in lead leaching from galvanic copper:lead solder coupons. The impact appeared to be due to non-uniform corrosion, as small pieces of solder were observed to detach into the water.

(3) In a water with a low concentration of nitrate (<2.25 mg/L NO₃–N) and disinfected with chloramine, corrosivity of the water to lead solder could increase due to changes in nitrogen speciation as would occur from chloramine decay and biological nitrification. Addition of chlorine and chloramine decreased lead leaching relative to a control condition without oxidants. These trends were altered in a water that already contained high nitrate (>5 mg/L NO₃–N).

Acknowledgments

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